SOP No. 28

Recommended Standard Operations Procedure for Using Advanced Weighing Designs

1 Introduction

1.1 Purpose

Advanced weighing designs use a combination of double substitution comparisons of weights of equal nominal value or a series of weights in ascending or descending order; standard(s), unknown weights, and an additional standard called a check standard. The weights are intercompared using an equal-arm, single-pan mechanical, full electronic, or a combination balance utilizing built-in weights and a digital indication. The specific SOP for the double substitution procedure for each balance is to be followed. Weighing designs provide two methods of checking the validity of the measurement using an F-test to check the measurement process and a t-test to evaluate the stability of the standard and check standard. Hence, the procedure is especially useful for high accuracy calibrations in which it is critical to assure that the measurements are valid and well documented. This procedure is recommended for precision calibration of laboratory working standards that are subsequently used for lower level calibrations, for routine calibration of precision mass standards used for calibration of other mass standards, and for surveillance of mass reference and working standards.

1.2 Prerequisites

1.2.1 Verify that valid calibration certificates are available for the standards used as restraints in the test.

1.2.2 Verify that the standards to be used have sufficiently small standard uncertainties for the intended level of calibration. Reference standards should only be used to calibrate the highest level of working standards in the laboratory and should not be used to routinely calibrate customer standards.

1.2.3 Verify that the balance used is in good operating condition with sufficiently small process standard deviation as verified by F-test values, pooled short term standard deviations, and by a valid control chart for check standards, or preliminary experiments to ascertain its performance quality when new balances are put into service. See NISTIR 5672[1] for a discussion on the performance levels expected for use of these procedures

[1]Fraley, K. L., Harris, Georgia G. L., NIST IR 5672, Advanced Mass Calibration and Measurement Assurance Program for State Calibration Laboratories, March 2005.

as part of a laboratory measurement assurance program to ensure traceability of laboratory standards.

1.2.4 Verify that the operator is experienced in precision weighing techniques, and has had specific training in SOP 2, SOP 4, SOP 5, SOP 29, and is familiar with the concepts in GMP 10. Further, the operator must have been trained in the creation of data files and the operation of the NIST Mass Code when it is used for data reduction as recommended. Example data sets and sample observation sheets are available in the Advanced Mass Seminar offered by the NIST Weights and Measures Division.

1.2.5 Verify that the laboratory facilities meet the following minimum conditions to meet the expected uncertainty possible with this procedure:

Table 1. Environmental conditions

Echelon	Temperature	Relative Humidity (percent)
I	20 °C to 23 °C, allowable variation of ± 1 °C maximum change of 0.5 °C/h	40 to 60 ± 5

2 Methodology

2.1 Scope, Precision, Accuracy

This method can be performed on any type of balance using the appropriate double substitution SOP for the particular balance. Because considerable effort is involved, this weighing design is most useful for calibrations of the highest accuracy. The weighing design utilizes a combination of double substitutions to calibrate a single unknown weight, or a group of related weights in a decade. This method introduces redundancy into the measurement process and permits two checks on the validity of the measurement; one on accuracy and stability of the standard and the other on process repeatability. A least-squares best fit analysis is done on the measurements to assign a value to the unknown weights. The standard deviation of the process depends upon the resolution of the balance and the care exercised to make the required weighings. The accuracy will depend upon the accuracy and uncertainty of the calibration of the restraint weights and the precision of the comparison.

2.2 Summary

A restraint weight, S, in some cases two restraint weights, S_1 and S_2, an unknown weight, X, or group of unknown weights, and a check standard, S_C are compared in a specific order typically using the double substitution procedure although other procedures may be appropriate. The balance and the weights must be prepared according to the appropriate double substitution SOP for the particular balance being used. Once the balance and weights have been prepared, all readings must be taken from the reading scale of the balance without adjusting the balance or weights in any way. Within a double substitution all weighings are made at regularly spaced time intervals to minimize effects due to instrument drift. Because of the amount of effort required to perform weighing designs, the procedure includes an air buoyancy correction using the average air density as determined immediately before and after the weighings, drift-free equation for calculating the observed differences, correction for the cubical coefficient of expansion when measurements are not made at 20 °C, an average sensitivity for the balance over the range of measurements made, and the international formula for air density.[2]

2.3 Apparatus/Equipment Required

2.3.1 Precision analytical balance or mass comparator with sufficient capacity and resolution for the calibrations planned.

2.3.2 Reference standard weights (usually starting at 1 kg or 100 g), calibrated check standards for each decade (e.g., 1 kg, 100 g, 10 g, 1 g, 100 mg, 10 mg, 1 mg for the seven series between 1 kg and 1 mg), working standard weights and sensitivity weights with valid calibrations traceable to NIST.

2.3.3 Small standard working standards with valid calibrations traceable to NIST to be used as tare weights. Note: The calculations performed by the mass code do not take into consideration the value of any tare weights used in the weighing design. Additional calculations will be required when tare weights are used.

2.3.4 Uncalibrated weights to be used to adjust the balance to the desired reading range or adequate optical or electronic range for the intended load and range.

2.3.5 Forceps to handle the weights or gloves to be worn if the weights are moved by hand.

2.3.6 Stop watch or other timing device to observe the time of each measurement or the operator is experienced with determining a stable indication. If an electronic balance is used that has a means for indicating a stable reading, the operator may continue to time readings to ensure consistent timing that can minimize errors due to linear drift.

2.3.7 Thermometer accurate to 0.10 °C with recent calibration certificate traceable to NIST to determine air temperature.[3]

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[2]Formula for the Density of Moist Air, (CIPM-81/91). This equation is published in SOP 2. The difference between Option A and Option B in SOP 2 is less than the uncertainty associated with air density equations.

[3]The thermometer, barometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement (See SOP 2).

2.3.8 Barometer accurate to 0.5 mm of mercury (66.5 Pa) with recent calibration certificate traceable to NIST to determine air pressure.

2.3.9 Hygrometer accurate to 10 percent with recent calibration certificate traceable to NIST to determine relative humidity.

2.3.10 Computer with sufficient processing capability and memory.

2.4 Procedure

2.4.1 Place the test weight and standards in the balance chamber or near the balance overnight to permit the weights and the balance to attain thermal equilibrium, or use a thermal soaking plate next to the balance with weights covered. Thermal equilibration time is particularly important with weights larger than 1 gram. An alternative heat source such as a heat lamp may further improve temperature stability in front of the balance. Conduct preliminary measurements to determine the size of the sensitivity weight and any tare weights that are required, adjust the balance to the appropriate reading range of the balance indications, and to exercise the balance. Refer to the appropriate double substitution SOP for details.

2.4.2 Weighing Designs

The table below shows the most common comparisons to be made as referenced in NBS Technical Note 952, Designs for the Calibration of Standards of Mass, J. M. Cameron, M. C. Croarkin, and R. C. Raybold, 1977. Each series is characterized by the number of observations, n, the degrees of freedom, *d.f.* associated with the standard deviation, the number of weights in each design, k (not shown in this table), the number of restraints (standards), and check standards, along with appropriate positions within the design*.

*Positions for check standards must be carefully considered as subsequent equations may be dependent on the position of use.

Table 2. Common weighing designs

Design ID	Description	*n* Observations	*d.f.* Degrees of freedom	Restraint Position	Check Std Position*
A.1.1	3-1 Weighing Design[4]	3	1	1	3
A.1.2	4-1 Weighing Design	6	3	1, 2	3 or 4
A.1.4	5-1 Weighing Design	10	6	1, 2	3,4, or 5
A.2.1	6-1 Weighing Design	8	3	1, 2	3,4,5, or 6
C.1**	5, 3, 2, 1, 1 Design (descending)	8	4	1, 2, 3	5 or 4
C.1	5, 3, 2, 1, 1 Design (ascending)	8	4	5	4
C.2	5, 3, 2, 1, 1, 1 Design (descending)	11	6	1, 2, 3	4,5, or 6
C.2	5, 3, 2, 1, 1, 1 Design (ascending)	11	6	6	4 or 5
C.9**	5, 2, 2, 1, 1 Design (descending)	8	4	1, 2, 3, 4	5
C.9	5, 2, 2, 1, 1 Design (ascending)	8	4	5	4
C.10	5, 2, 2, 1, 1, 1 Design (descending)	8	3	1, 2, 3, 4	5 or 6
C.10	5, 2, 2, 1, 1, 1 Design (ascending)	8	3	6	4 or 5

**If these designs are NOT the last in a series, there is no position for a check standard.

The "restraint" is another name for the standard used in the comparison. Matrices are shown in Technical Note 952. Determine the best design prior to beginning the series. The series shown allow calibration of any commonly found set of mass standards in either the 5, 2, 2, 1 combination or the 5, 3, 2, 1 combination.

2.4.3 Measurement Procedure

Record the pertinent information for all weights being intercompared on a suitable data sheet unless an automated data collection system is being used to collect the data and create a data file. Record or collect the laboratory ambient temperature, barometric pressure, and relative humidity immediately before and immediately after each series of intercomparisons.

3 Calculations

Calculations are completed by the NIST Mass Code as described in NBS Technical Note 1127, National Bureau of Standards Mass Calibration Computer Software, R. N. Varner, and R. C. Raybold, July 1980, with updates to conform to the international formula for calculating air density and the ISO Guide to the Expression of Uncertainties, 1993, and

[4]Design a.1.1. with inverted order (y3, y2, and y1), with restraint in position 1 (B) is detailed in SOP 5.

minor error corrections to the original code. The code is the same as that used by the NIST Mass Group for routine calibrations. The code performs two statistical tests (t-test and F-test) to verify both the value of the restraints and check standards, and to verify that the measurement process was in control during the comparisons.

3.1 Calculating Effective Densities and Coefficients of Expansion for Summations[5]:

Some designs use a summation mass and sometimes the individual masses of this summation will be constructed from different materials that have different densities and coefficients of expansion. The following equations will be used to calculate the effective density and effective coefficient of expansions for the summation that will be needed as input for the data file. The subscripts 5, 3, and 2 refer to the individual masses that comprise the summation. This approach may also be needed with a 5, 2, 2, 1 combination.

$$Effective\ Density = \frac{M_5 + M_3 + M_2}{\left(\frac{M_5}{\rho_5}\right) + \left(\frac{M_3}{\rho_3}\right) + \left(\frac{M_2}{\rho_2}\right)}$$

$$Effective\ Cubical\ Coefficient\ of\ Expansion = \frac{\left(\frac{M_5}{\rho_5}\alpha_5\right) + \left(\frac{M_3}{\rho_3}\alpha_3\right) + \left(\frac{M_2}{\rho_2}\alpha_2\right)}{\left(\frac{M_5}{\rho_5}\right) + \left(\frac{M_3}{\rho_3}\right) + \left(\frac{M_2}{\rho_2}\right)}$$

Table 3. Variables for equations above

Variable	Description
M	Mass (g)
ρ	Density (g/cm^3)
α	Cubical Coefficient of Expansion (/°C)

4 Assignment of Uncertainty

The NIST Mass Code generates uncertainties as a part of the data reduction. Proper input in the data file is critical for obtaining valid results and is dependent upon a well characterized measurement process. See NIST IR 5672 for a discussion on the input for standard uncertainties in the data file.

4.1 Calculating the standard uncertainty, u_S, of the starting restraint in the first series:

Usually the starting restraint will be one or several 1 kg (or 100 g) mass standards that have NIST calibrations and density determinations. The uncertainty of the

[5]Jaeger, K B., and R. S. Davis, NIST Special Publication 700-1, A Primer for Mass Metrology, November 1984.

standard as stated on a calibration report is divided by two or three, dependent on the confidence interval stated in the calibration report.

One starting restraint scheme (a single starting standard), where U_S is the uncertainty from NIST which must be divided by the proper coverage factor, k.

$$u_s = \frac{U_s}{k_{factor}}$$

Multiple starting restraint scheme with standards calibrated at the same time against the same starting standards, i.e., dependent calibration (more than one starting standard):

$$u_s = \frac{U_{s1}}{\mathrm{k}_{\mathrm{factor1}}} + \frac{U_{s2}}{\mathrm{k}_{\mathrm{factor2}}},$$

or

$$u_s = \frac{U_{s1}}{\mathrm{k}_{\mathrm{factor1}}} + \frac{U_{s2}}{\mathrm{k}_{\mathrm{factor2}}} + \frac{U_{s3}}{\mathrm{k}_{\mathrm{factor3}}}, \quad etc.$$

Multiple starting restraint scheme with standards *NOT* calibrated at the same time as the starting standards, i.e., independent calibration (more than one starting standard):

$$u_s = \sqrt{\left(\frac{U_{s1}}{\mathrm{k}_{\mathrm{factor1}}}\right)^2 + \left(\frac{U_{s2}}{\mathrm{k}_{\mathrm{factor2}}}\right)^2},$$

or

$$u_s = \sqrt{\left(\frac{U_{s1}}{\mathrm{k}_{\mathrm{factor1}}}\right)^2 + \left(\frac{U_{s2}}{\mathrm{k}_{\mathrm{factor2}}}\right)^2 + \left(\frac{U_{s3}}{\mathrm{k}_{\mathrm{factor3}}}\right)^2}, \quad etc.$$

4.2 Calculating the within-process standard deviation, s_W, for a particular series:

For each particular weighing design, the observed within process standard deviation, s_w, along with its degrees of freedom, d.f., is pooled using the technique described in NIST Handbook 145 section 8.4.

$$s_w = \sqrt{\frac{(df_1)s_1^2 + (df_2)s_2^2 + ... + (df_k)s_k^2}{df_1 + df_2 + ... + df_k}}$$

4.3 Calculating the between-time standard deviation for each particular series (s_b):

Establish a standard deviation (s_t) for each check standard over time. If a plot of the check standard shows no apparent drift, the between-time standard deviation may be calculated. The following formulae are used to calculate the between-time standard deviation for the particular series. If s_b^2 is less than zero, then s_b equals zero.

4.3.1 For the 3-1 design with a single restraint, and a check standard that is either another single weight or a summation, the between time standard deviation is calculated using the following formula. The check standard may be in any position.

$$s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$

$$K_1 = 0.8165$$

$$K_2 = 1.4142$$

$$s_b = \frac{1}{1.4142}\sqrt{s_t^2 - 0.8165^2 s_w^2}$$

4.3.2 Using a 4-1 design with two restraints, and the check standard is the difference between the two restraints, the next equation may be used to calculate the between-time standard deviation. If another weight in the series is used as the check standard, another equation is needed.

$$s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$
$$K_1 = 0.7071$$
$$K_2 = 1.4141$$
$$s_b = \frac{1}{1.4141}\sqrt{s_t^2 - 0.7071^2 s_w^2}$$

4.4.3 Using a 4-1 design with two restraints, and with a single check standard occupying any of the remaining positions, the next equation may be used to calculate the between-time standard deviation.

$$s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$
$$K_1 = 0.6124$$
$$K_2 = 1.2247$$
$$s_b = \frac{1}{1.2247}\sqrt{s_t^2 - 0.6124^2 s_w^2}$$

4.3.4 Using a 5-1 design with two restraints, and the check standard is the difference between the two restraints, the next equation may be used to calculate the between-time standard deviation. If another weight in the series is used as the check standard, another equation is needed.

$$s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$
$$K_1 = 0.6325$$
$$K_2 = 1.4142$$
$$s_b = \frac{1}{1.4142}\sqrt{s_t^2 - 0.6325^2 s_w^2}$$

4.3.5 Using a 5-1 design with two restraints, and with a single check standard occupying any of the remaining positions, the next equation may be used to calculate the between-time standard deviation.

$$s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$
$$K_1 = 0.5477$$
$$K_2 = 1.2247$$
$$s_b = \frac{1}{1.2247}\sqrt{s_t^2 - 0.5477^2 s_w^2}$$

4.3.6 In the second series (C.2), six weights are involved (500 g, 300 g, 200 g, 100 g, Check 100 g, and a summation 100 g). Calculate the standard deviations of the mass values for the Check 100 g (s_t) and plot the results to evaluate the presence or lack of drift. If no drift is present, the following formula is used to calculate the between-time standard deviation for this series and all subsequent C.2 series. Subsequent series include the following check standards: 100 g, 10 g, 1 g, 100 mg, 10 mg, 1 mg. If s_b^2 is less than zero, then s_b equals zero.

$$s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$

$$K_1 = 0.3551$$

$$K_2 = 1.0149$$

$$s_b = \frac{1}{1.0149}\sqrt{s_t^2 - 0.3551^2\, s_w^2}$$

4.3.7 If a C.1 series is used, the following equation is used to calculate the between-time standard deviation when the check standard is in either of the last two positions:

$$s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$

$$K_1 = 0.4253$$

$$K_2 = 1.0149$$

$$s_b = \frac{1}{1.0149}\sqrt{s_t^2 - 0.4253^2\, s_w^2}$$

4.3.8 The between-time formulae shown here are those that are most common and are for descending series only. If another restraint or check standard is used, or if an ascending series is used, another formula will be needed. These formulae are statistically derived, based on the least squares analysis of the weighing design, and assume a normal, non-drifting distribution of measurement results. Equations for some other weighing designs may be calculated using the NIST Electronic Engineering Statistics Handbook. Section 2.3.3.2 “Solutions to Calibration Designs” gives an overview for deriving the solutions to weighing designs. It also provides the unifying equation for s_b (it is called s_{days} in the electronic handbook). To clarify the difference in terminology and notation the unifying equation for s_b is presented as:

$$s_{days} = \frac{1}{K_2}\sqrt{s_2^2 - K_1^2 s_1^2} \qquad \begin{aligned} s_{days} &\equiv s_b \\ s_1 &\equiv s_w \\ s_2 &\equiv s_t \end{aligned} \qquad s_b = \frac{1}{K_2}\sqrt{s_t^2 - K_1^2 s_w^2}$$

Section 2.3.4.1 “Mass Weights” provides the solutions for 17 weighing designs used for decreasing weight sets, 6 weighing designs for increasing weight sets and 1 design for pound weights. K_1 is located in the portion of the solution titled “Factors for Repeatability Standard Deviations”, and K_2 is located in the portion titled “Factors for Between-Day Standard Deviations”.

5 Report

Report results as printed in Tables I and II as generated by the Mass Code. Actual text of the mass code report must be modified for each laboratory in order to be ISO/IEC 17025 compliant.